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The microstructural difference between weakly aggregated ("dispersed") and strongly aggregated clays means that a larger part of the water is structurally unaffected by clay mineral lattices in the last-mentioned type of clay which should therefore have a lower content of unfrozen water. This hypothesis was tested and found to be valid in an investigation where two such clays were actually compared with reference to the content of unfrozen water. The results from dilatometer and spin echo tests clearly show that this content is ──── CONT

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dependent not only on the specific surface area but also on the microstructural constitution expressed in terms of the degree of aggregation.

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CLAY MICROSTRUCTURE AND ICE NUCLEATION [1]

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[1] In this report ice nucleation stands for the formation of ice at very moderate negative temperatures in clays under closed conditions.

CLAY MICROSTRUCTURE AND ICE NUCLEATION

SUMMARY

The microstructural difference between weakly aggregated
("dispersed") and strongly aggregated clays means that a
larger part of the water is structurally unaffected by clay
mineral lattices in the last-mentioned type of clay which
should therefore have a lower content of unfrozen water.
This hypothesis was tested and found to be valid in an in-
vestigation where two such clays were actually compared
with reference to the content of unfrozen water. The results
from dilatometer and spin echo tests clearly show that
this content is dependent not only on the specific surface
area but also on the microstructural constitution expressed
in terms of the degree of aggregation.

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INTRODUCTION, SCOPE OF INVESTIGATION

The main characteristic feature of illite clay microstruc-
ture is aggregation, which is very strong in marine clays
but less obvious in fresh water-deposited clays. Aggrega-
tion means that the particles form fairly stable groups
(secondary particles) separated by voids. In principle,
the microstructural difference between fresh water clays
and marine clays is as shown in Fig. 1 and this has also
been confirmed by a number of investigators (cf. PUSCH 1970,
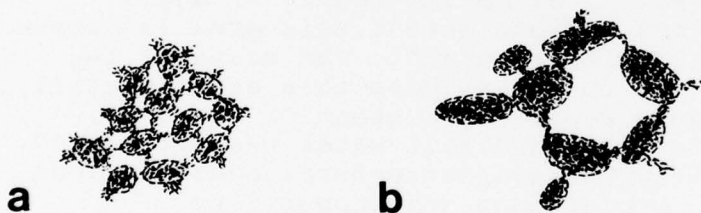BARDEN 1973).

Fig. 1   Schematic picture of the microstructural pattern
         of natural illitic clay a) deposited in fresh
         water, b) deposited in salt water.

Fig. 1 means that the particles in fresh-water clay are
collected in a large number of small but fairly porous aggre-
gates separated by small voids, while they form big and
dense aggregates separated by large voids in marine clay.
A larger fraction of the soil water is located within the
very small intra-aggregate voids in weakly aggregated ("dis-
persed"[1]) than in strongly aggregated clays. This can also

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[1] This description is commonly used for clays with a fairly
     uniform particle distribution. There is no single particle
     behaviour, however.

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be expressed by saying that a major part of the water is structurally unaffected by clay mineral lattices in the last-mentioned typ of clay. The consequence is that a weakly aggregated clay should have a higher percentage of unfrozen water than a strongly aggregated clay with the same electrolyte, mineralogical and granulometrical compositions. Ice nucleation should occur more easily and rapidly in the "free water" in the large voids of strongly aggregated clay.

Previous investigations (PUSCH, 1977) support this hypothesis. Thus, electron microscopical (SEM) studies on freezing clay have shown that many particle aggregates are moved without much internal distortion, which indicates that much of the intra-aggregate water does not freeze in the temperature interval 0° to $-5^{\circ}C$. The conclusion of this work was that the amount of unfrozen water should be large when the particle aggregates are small and fairly porous but closely spaced since a considerable mineral surface is then exposed to the water. In principle, this is in agreement with the statement by DILLON & ANDERSLAND (1966) and ANDERSON & TICE (1972) that the unfrozen water is a function of the specific surface area. However, since the degree of aggregation in fact determines to what extent this area is exposed to water it should be equally important. The scope of the present investigation was to investigate this experimentally. For this purpose the unfrozen water content of two soft, illitic clays formed in fresh and salt water was determined. The clays had quite different microstructural constitutions but otherwise similar compositions and properties.

CLAY TYPES

Geotechnical properties

A fresh-water clay from Skå-Edeby (Stockholm area) and a marine clay from Lilla Edet (Swedish west-coast) were used for the investigation. Their main geotechnical properties are given in Table 1. The sampling depth was 5 m at the Skå-Edeby and 3 m at the Lilla Edet site.

The fresh-water clay from Skå-Edeby is normally sensitive while the marine clay from Lilla Edet is quick. The high sensitivity of the latter clay is because it has been leached by percolating fresh water in nature.

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Table 1 Geotechnical data

| Clay | ρ | τ_{fu} | S_t | w | w_L | w_P | Organic content |
	t/m³	kPa		%	%	%	%
Skå-Edeby	1.42	8-10	12	82-106	93	29	~1
Lilla Edet	1.48	13-18	42	97-101	71-73	33	~1

Grain size distribution

The plastic limit is of the same order of magnitude for the two clays while the Skå-Edeby clay has a somewhat higher liquid limit which indicates a higher clay content (Table 2).

Table 2 Grain size distribution

| Clay | Weight percentage of material finer than | | | | |
	0.04 mm	0.02 mm	0.01 mm	0.006 mm	0.002 mm
Skå-Edeby	82	80	79	79	77
Lilla Edet	99	99	90	88	67

Previous electron microscopical investigations have shown that the average particle size of the clay fraction is much smaller in the Lilla Edet clay than in the Skå-Edeby clay (PUSCH, 1970). Thus, using the definition of particle size given by Fig. 2, it was found that the median value **a** was only 0.018 μm for the first-mentioned clay while it was 0.037 μm for the Skå-Edeby clay. A microstructural study showed that a large number of particles smaller than 0.02 μm act as linking units in the particle network. Hence, they were not formed after the deposition of the sediment and they are not a product of the preparation of the clay suspensions for the granulometrical investigation. A detailed description of the granulometrical and morphological properties of the clay fractions is given in Table 3.
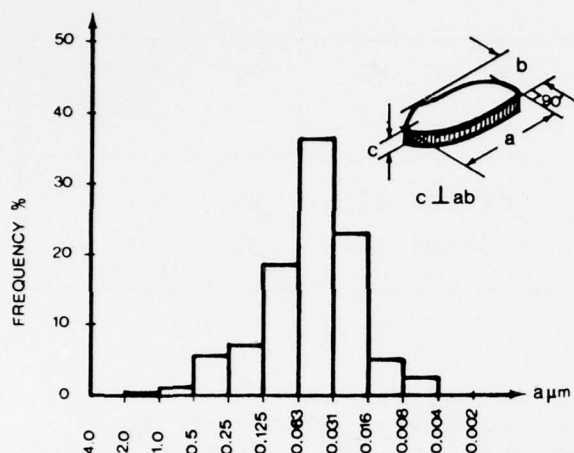
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Fig. 2　Example of histogram of particle size for the clay
fraction.

Table 3　Median values, quartiles　and percentiles of **a** and
a/c for clay particles

Clay	**a** in μm						**a/c**		
	M^1	$Q_1{}^2$	$Q_3{}^3$	$P_{10}{}^4$	$S_K{}^5$	$\log S_0{}^6$	M	Q_1	Q_3
Skå-Edeby	0.037	0.026	0.083	0.021	1.58	0.252	4.0	2.9	5.9
Lilla Edet	0.018	0.014	0.025	0.012	1.08	0.126	2.7	1.8	3.3

[1] Median value
[2] Lower quartile
[3] Upper quartile
[4] 10th percentile (finest part of the cumulative curve)
[5] Skewness (TRASK 1932), $(Q_1 \cdot Q_3)/M^2$
[6] S_0 = sorting coefficient $\sqrt{Q_3/Q_1}$; log S_0-values are direct-
ly comparable

The presence of many particles of colloidal size in the
clay fraction of the Lilla Edet clay is of great importance
as concerns the specific surface area. As will be shown
later in the text this means that although the clay content
(weight percentage of material finer than 2 μm) is higher
for the Skå-Edeby clay, the specific surface　area is much
higher for the Lilla Edet clay.

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Mineralogy

X-ray diffraction analyses (cf. Fig. 3) of particles finer
than 10 μm have shown that the main minerals in the Skå-
Edeby clay are carbonate minerals, (mainly calcite), illite,
quartz, kaolinite, feldspars and chlorite, the three first-
mentioned being dominant. In the Lilla Edet clay, which is
rich in rock flour, the main minerals are feldspars, quartz,
illite, kaolinite, hornblende and rock-forming micas. Illite
is the dominant clay mineral in both clays. The clay fraction
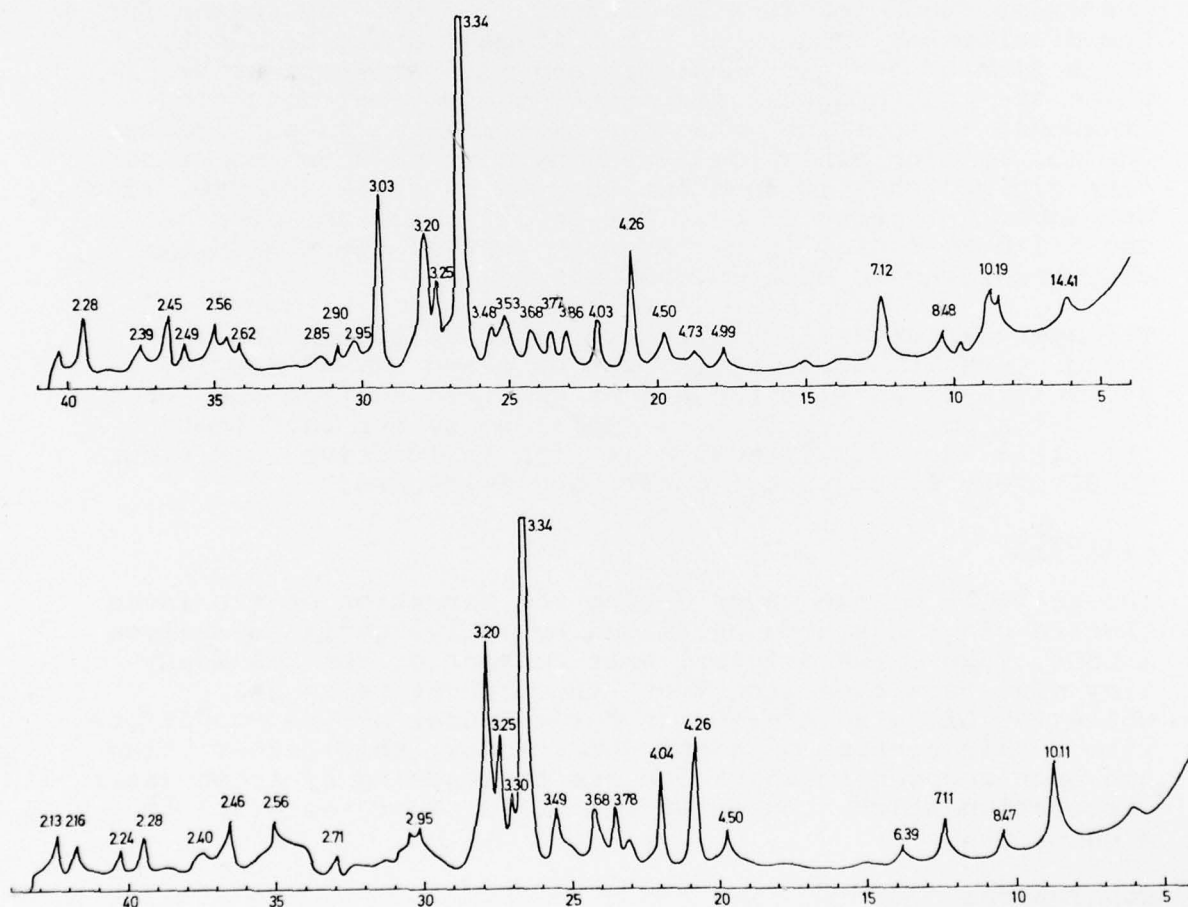of the Lilla Edet clay contains considerable amounts of
quartz and feldspars.

Fig. 3   Representative diffractometer records showing d-
         values (horizontal axis gives 2θ°). Upper diagram
         Skå-Edeby. Lower diagram Lilla Edet.

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Specific surface area

The specific surface area was determined in two ways. Firstly,
it was calculated on the basis of Table 3 assuming the par-
ticles to have a cylindrical shape with a diameter equal to
a and a height equal to **C** . The calculation gave 99 m²/g for
the Skå-Edeby clay and 204 m²/g for the Lilla Edet clay.
Secondly, it was determined experimentally by applying the
BET method and MADSEN's (1977) glycerol sorption technique.
This investigation gave 690 m²/g for the Skå-Edeby clay and
996 m²/g for the Lilla Edet clay. The latter values, which
should be more reliable than the theoretically deduced ones,
indicate rather extreme specific surface areas, almost of
the order found for 100% Na-montmorillonite. The reason for
the discrepancy is not known but it may partly be due to
the action of organic substance and mineral wheathering
which are not accounted for in the theoretical deduction.
It should be mentioned that the experimental values are not
improbably high since the Skå-Edeby clay is known to be so
fine-grained that it does not obey Darcy's law (HANSBO, 1960).
The important thing is that the specific surface area of
the Lilla Edet clay is higher than that of the Skå-Edeby
clay according to both determinations.

The average surface activity expressed by the cation ex-
change capacity is similar for both clays (29-33 meq/100 g
dry material) despite the higher specific surface area of
the Lilla Edet clay. This is explained by the fact that
the Lilla Edet clay fraction is rich in inactive rock flour,
mainly very fine-grained quartz and feldspars.

Salinity

The salinity of the water during the formation of the inves-
tigated clays has been estimated by applying diatom analyses
(PUSCH, 1970). The original salt content of the Skå-Edeby
clay must have been less than the present value 5‰,
while the Lilla Edet clay was formed under marine conditions
with a salt content of about 35‰ . Today, this latter value
has been reduced to about 5‰ due to leaching by fresh-water
percolation which converted the Lilla Edet material to
a quick clay.

Conclusions

The two clays have quite different microstructural constitu-
tions but otherwise similar compositions and properties
except for the specific surface area, which is higher for
the marine Lilla Edet clay. This should give the Lilla Edet
clay the highest content of unfrozen water if there were no in-
fluence of the microstructural geometry. A comparison bet-
ween the contents of unfrozen water of the clays can there-

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fore tell us whether this geometry - or rather the degree
of aggregation - has any effect.

TESTS

Equipment

Dilatometer technique was applied for the determination of
the amount of unfrozen water. The samples (40 mm diameter
and 20-25 mm height) were mounted in closely fitting steel
cylinders which allowed uni-axial swelling only and no uptake
of external water (closed system). The design of the dila-
tometer is shown in Figs. 4 and 5.

The cylinder, which was equipped with a sharp edge, was
pushed into the clay to a depth of 20-25 mm. After truncation
with a thin steel wire the lids were applied and the device
closed. This operation was made in a batch with paraffine
oil (purum), which was the liquid used for measuring the
volume changes. Since the whole device was submerged in the
oil throughout the mounting operation there should be no
air left in the cylinder and glass pipe or any other part
of the measuring system. Thus, by calculating the temperature-
dependent volume change of the steel cylinder and by deter-
mining experimentally the same property of the paraffine
oil, the device was calibrated so that an observed displace-
ment of the oil meniscus in the glass pipe could be expressed
in terms of a volume change of the clay sample. The rela-
tionship turned out to be 29.3 mm^3 per mm displacement.
The latter could be measured with an accuracy of ±0.01 mm.
Then, the amount of ice could be estimated as a function of
temperature by assuming that water expands 9.1% when it
freezes.

Two cells were mounted on a frame with a precision device
(metal scale and lens) for measuring the temperature-depen-
dent displacement of the oil meniscus (Fig. 6).

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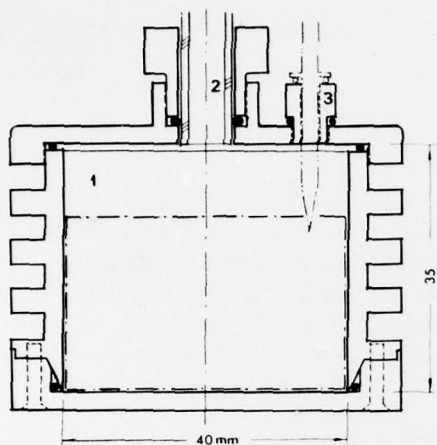CLAY MICROSTRUCTURE AND ICE NUCLEATION

Fig. 4 Section through dilatometer.
1. Steel cylinder for clay sample and paraffine oil.
2. Glass pipe for measuring the volume of extruded paraffine oil.
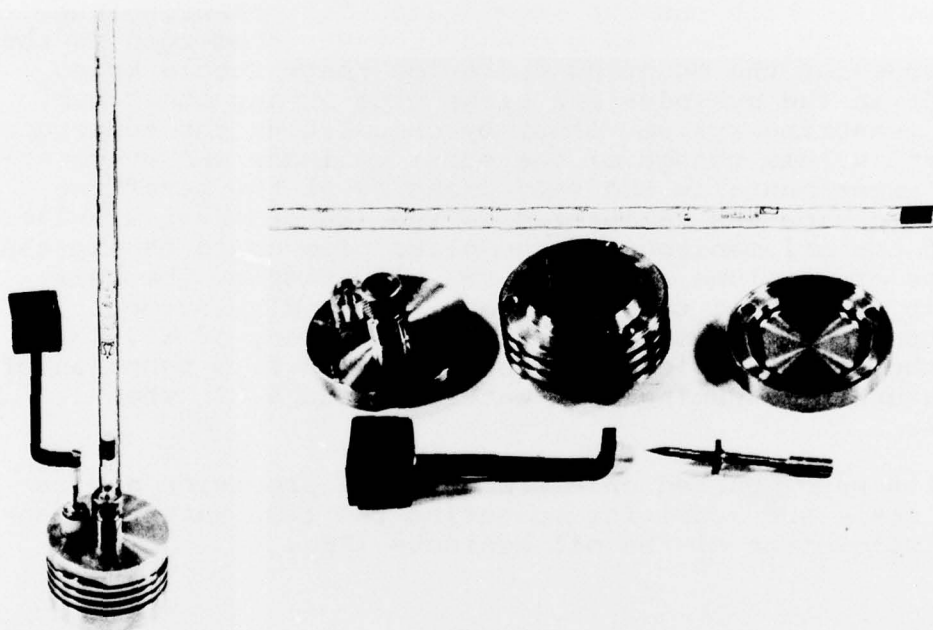3. Insulation of plastic with copper needle for ice nucleation.

Fig. 5 The dilatometer. Left picture shows assembled device. Note the cup and needle for liquid air cooling (ice nucleation) in the right picture which shows the various components.
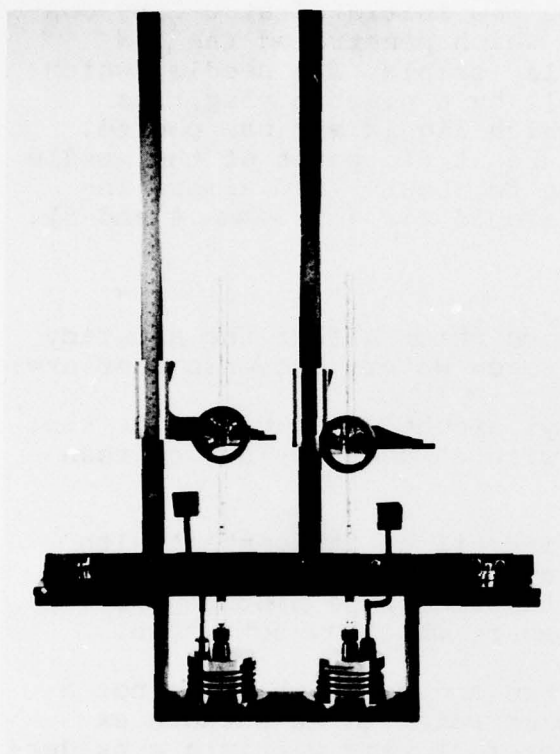
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Fig. 6   Frame with two cells ready to be submerged in the
         methyl alcohol bath.

The frame was then submerged in a Hetofrig CB-6 cooling de-
vice with methyl alcohol in its cooling chamber. Insula-
tion material was applied around the glass pipe and the
metal pieces extending from the chamber so that a uniform
temperature of all the parts of the measuring device was ob-
tained.

Program

The temperature was lowered stepwise to ± 0, -1, -2, -3, -4,
-5, -6, -7, -8, -9, and $-10^{O}C$ and was then raised again in
$1^{O}C$ steps until $\pm 0^{O}C$ was reached and the samples thawed.
Equilibrium after each temperature change was obtained with-
in 3-6 hours.

Tests were made with undisturbed as well as with remoulded
samples. To avoid the entrance of air into the samples at

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the remoulding they were wrapped in thin plastic bags. They were then pressed and gently kneaded.

In all the tests ice nucleation was initiated at 0°C by cooling a very fine copper needle which penetrated the lid and a few millimeters of the clay sample. The needle, which was isolated from the metal cell by a plastic plug, was equipped with a small cup in which liquid air was poured. It was found that the temperature at the point of the needle decreased from room temperature to about -70°C almost instantly at the application of liquid air (cf. Figs. 4 and 5).

Accuracy

There are several possible errors which affect the accuracy of the measured content of unfrozen water. The main ones are:

1. The temperature of the methyl alcohol bath cannot be kept at the desired temperature with an accuracy better than $\pm 0.1^\circ$C.

2. The behaviour of the paraffine oil at its contact with the clay at sub-zero temperatures is not known. There may be a zone where water and oil form a mixture the temperature/volume properties of which are not known.

3. The ice formed in the clay has a density which is not a constant. It contains unfrozen water to an unknown extent and its density can therefore vary within a considerable interval.

The last-mentioned point is most important and it was concluded that the amount of unfrozen water can be determined with an accuracy of no better than about $\pm 10\%$ when applying the technique described here. This was considered to be sufficiently accurate for the present purpose. It was decided, however, to check the results by applying a different technique. This check, which is described later in the text, confirmed the main conclusions from the dilatometer tests.

Test results

Fig. 7 shows the unfrozen water content (w_u=ratio of unfrozen water mass and mass of solid substance) as a function of temperature for undisturbed samples.

The two curves show the relationship for a successively lowered temperature (0°C and lower). We see that the unfrozen water content of the Skå-Edeby clay is considerably higher than that of the Lilla Edet clay. Thus, the hypothesis that the "dispersed" fresh-water clay should have a higher percentage of unfrozen water than the leached marine clay is valid.
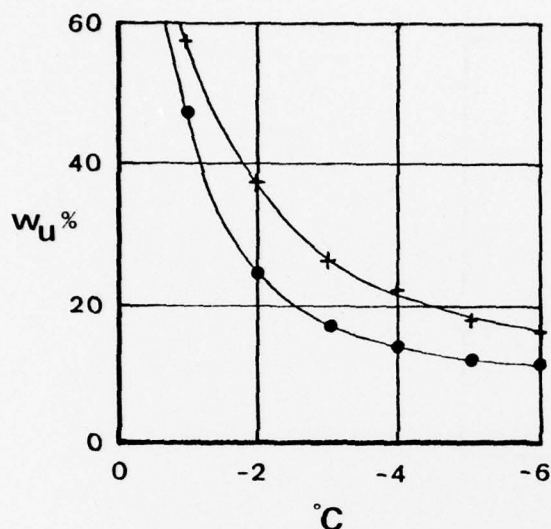
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Fig. 7   Undisturbed Skå-Edeby (+) and Lilla Edet (●) samples.

This hypothesis implies that if a structural change is
caused so that the two clays become identical with respect
to the degree of aggregation, they will also have the same
amount of unfrozen water. Such a structural change can be
obtained by remoulding (Fig. 8). Fig. 9, which shows the
results from tests with remoulded samples, confirms that
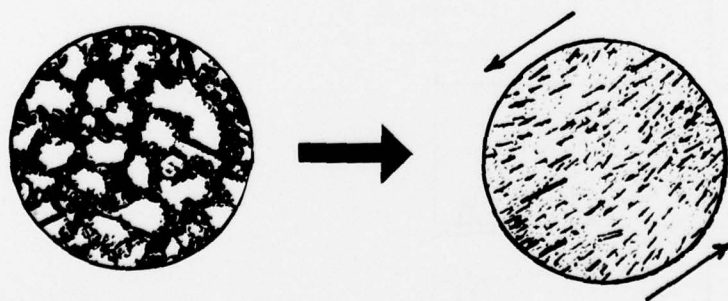practically identical amounts of unfrozen water are obtained.

Fig. 8   Schematical picture of the microstructural change
          induced by large shear strain or remoulding
          (ARNOLD, 1967).
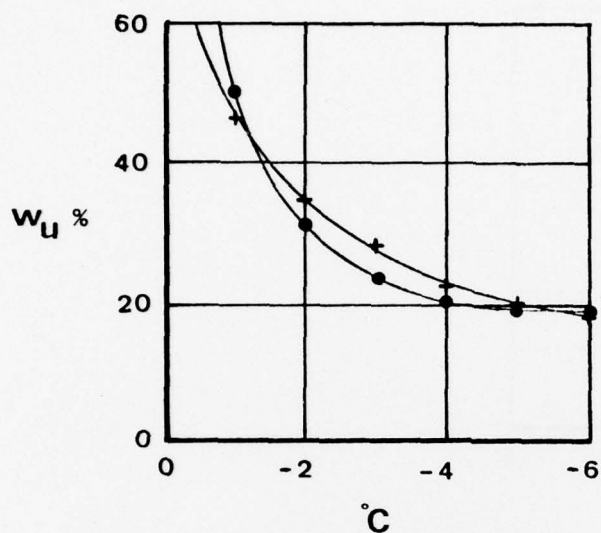
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Fig. 9   Remoulded Skå-Edeby (+) and Lilla Edet (●) samples.

The explanation is of course that the remoulding produces particle dispersion which increases the mineral surface exposed to pore water and thereby also the amount of unfrozen water just as we can see from Fig. 10.
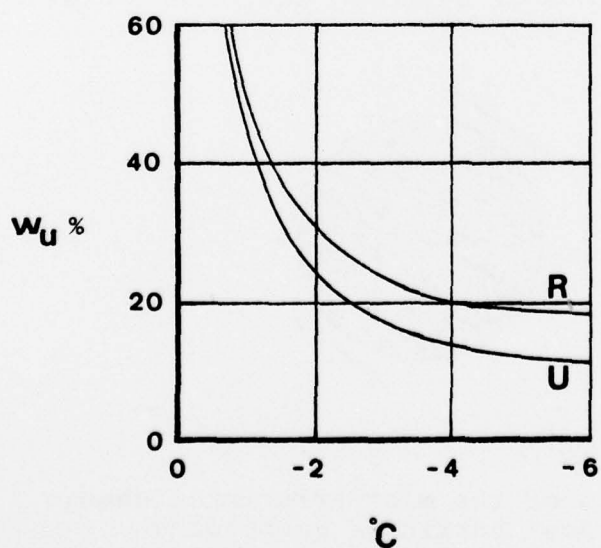

Fig. 10   Lilla Edet samples. U = undisturbed, R = remoulded.

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In fresh-water clays with a high degree of particle dis-
persion, remoulding may improve the dispersion a little
but it will also create a certain number of larger continuous
pores. Since the majority of the water in such pores is
readily frozen the net effect will be a slightly reduced
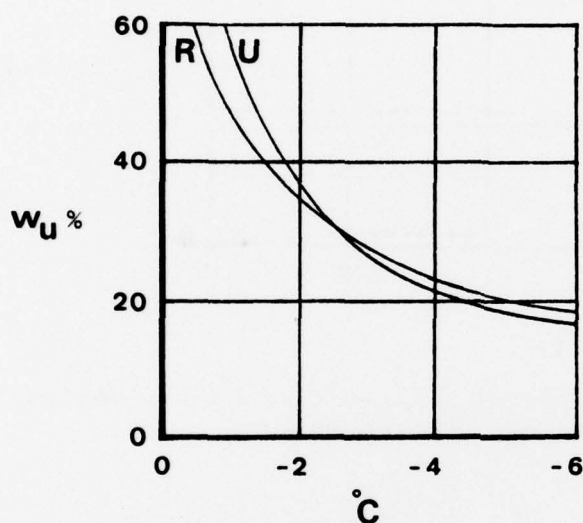amount of unfrozen water in the vicinity of $0^{\circ}C$ (Fig. 11).

Fig. 11   Skå-Edeby samples. U = undisturbed, R = remoulded

The consolidation effects are very well illustrated by the
hysteresis loops in Fig. 12. This picture shows that when
temperature is lowered from $-1^{\circ}C$ to $-10^{\circ}C$ and then increased
again to $-1^{\circ}C$, the unfrozen water was reduced to about half
the original amount. This is because consolidation produced
zones or regions which behaved as very big, dense and
fairly "dry" particle aggregates between ice accumulations.
The water transported to freezing zones in connection with
the formation of consolidated big aggregates (Fig. 13) is
not readily brought back into the dense aggregates when the
temperature increases and a net decrease in the amount of
adsorbed water is therefore obtained.

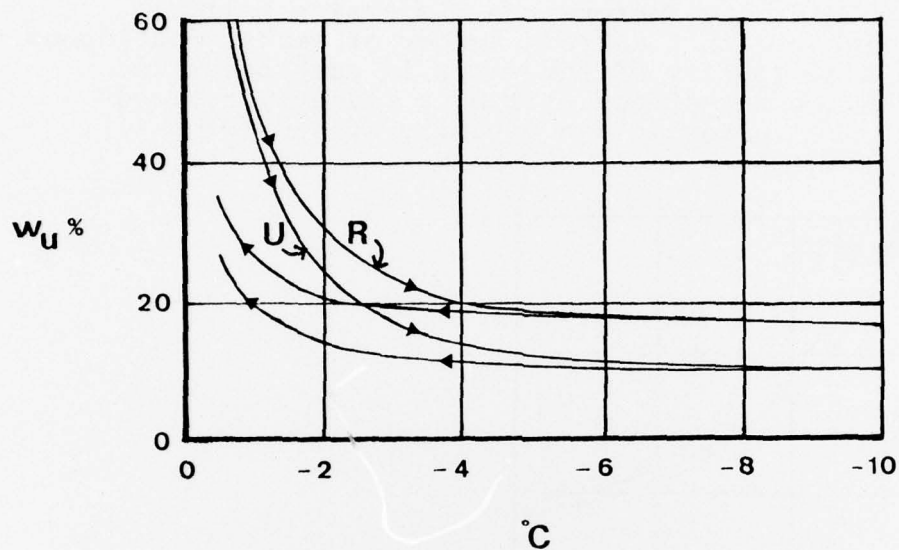CLAY MICROSTRUCTURE AND ICE NUCLEATION

Fig. 12 Lilla Edet samples frozen to -10°C and then heated to 0°C. U = undisturbed, R = remoulded.

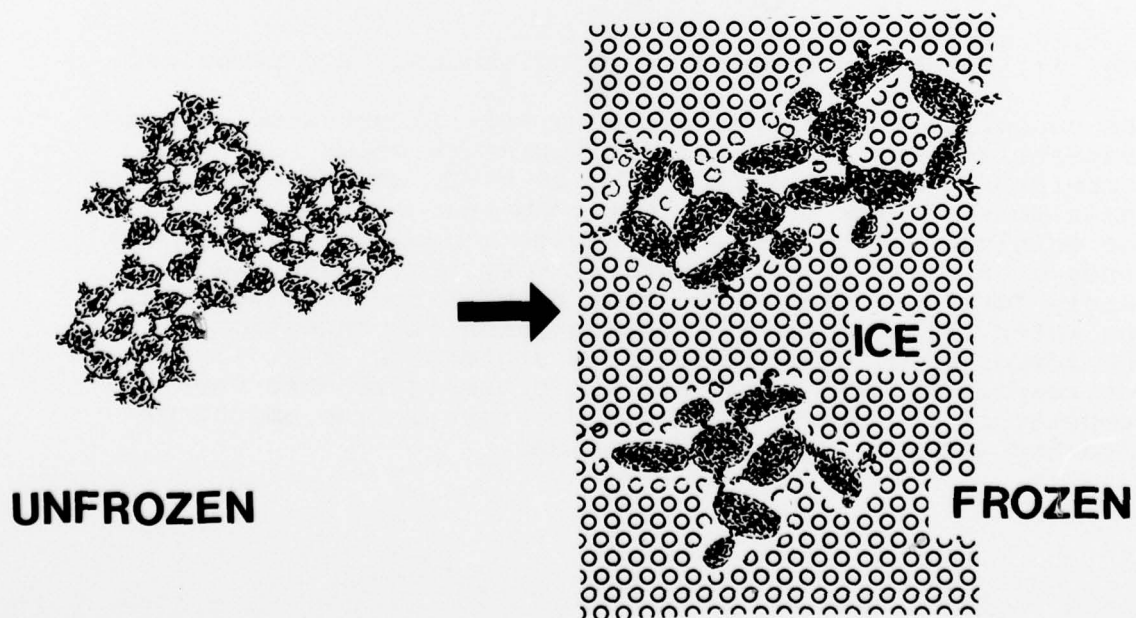UNFROZEN   ICE   FROZEN

Fig. 13 Local consolidation produced by freezing.

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Spin echo technique test

Pulsed NMR technique has been applied at the U S Army Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire, USA, for several years and a practical method of determining the phase composition of frozen soil (unfrozen water content vs temperature) has been described recently by TICE, BURROUS and ANDERSON (1978). Samples of the Skå-Edeby and Lilla Edet clays were sent to CRREL[1] for determination of the amount of unfrozen water.

Representative results of the CRREL study are given in Tables 4 and 5 together with the author's results.

Table 4 Unfrozen water content of undisturbed Skå-Edeby clay

Temperature $^{\circ}C$	w_u% CRREL	w_u% AUTHOR
-1.0	47	57
-2.0	31-35	38
-3.0	26-34	26
-4.0	22-24	22
-5.0	20-21	18
-6.0	18-19	16

Table 5 Unfrozen water content of undisturbed Lilla Edet clay

Temperature $^{\circ}C$	w_u% CRREL	w_u% AUTHOR
-1.0	36	47
-2.0	25	24.5
-3.0	20-21	18
-4.0	17-18	15
-5.0	15	12
-6.0	14	11

[1] Part of a current research cooperation.

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Tables 4 and 5 show that the agreement is good for the w_u-values determined by the two methods, especially in the temperature interval -2 to -4°C. It is concluded also that the CRREL tests confirm the author's result that the Skå-Edeby clay has a higher amount of unfrozen water than the Lilla Edet clay at each specific temperature.

MEANS OF DETERMINING AND EXPRESSING THE DEGREE OF AGGREGATION

At least for scientific purposes it should be possible to apply a method used for classifying clay microstructure to determine and express the degree of aggregation (PUSCH, 1970). It requires that the clay is impregnated and that ultra-thin sections are cut by applying ultra-microtomy, and that elect-ron micrographs (TEM) are taken.

Micrographs obtained from ultra-thin sections (300 Å) repre-sent two-dimensional cross sections through the clay and this offers a possibility of defining microstructural para-meters and applying simple statistical methods to describe the patterns. The sectioned pore area P in percent of the total area T of the micrograph is a suitable parameter, which can be termed "two-dimensional porosity". The measurement of these quantities is made by using drawn images of the micrographs. In these drawings the pore area is marked white while particles and aggregates are marked black regardless of their density (Fig. 14).

For homogeneous clay it has been shown that this parameter, determined by using 5-10 micrographs, is representative of volumes of several cubic centimeters although the ana-lyzed sections are very small. The very obvious difference in the degree of aggregation seen in Fig. 14 is illustrated by the P/T-values. Thus, fig. 15 shows P/T for the two salinity regions which are representative of the Skå-Edeby (lower histogram) and Lilla Edet (upper histogram) clays.

There is no simple relationship between P/T, the specific surface area and the amount of unfrozen water. It is reason-able to believe, however, that P/T or some function of it can be used to correct the specific surface area which is a basic measure of the unfrozen water content. The matter requires additional work before a reliable correction fac-tor can be suggested, however.

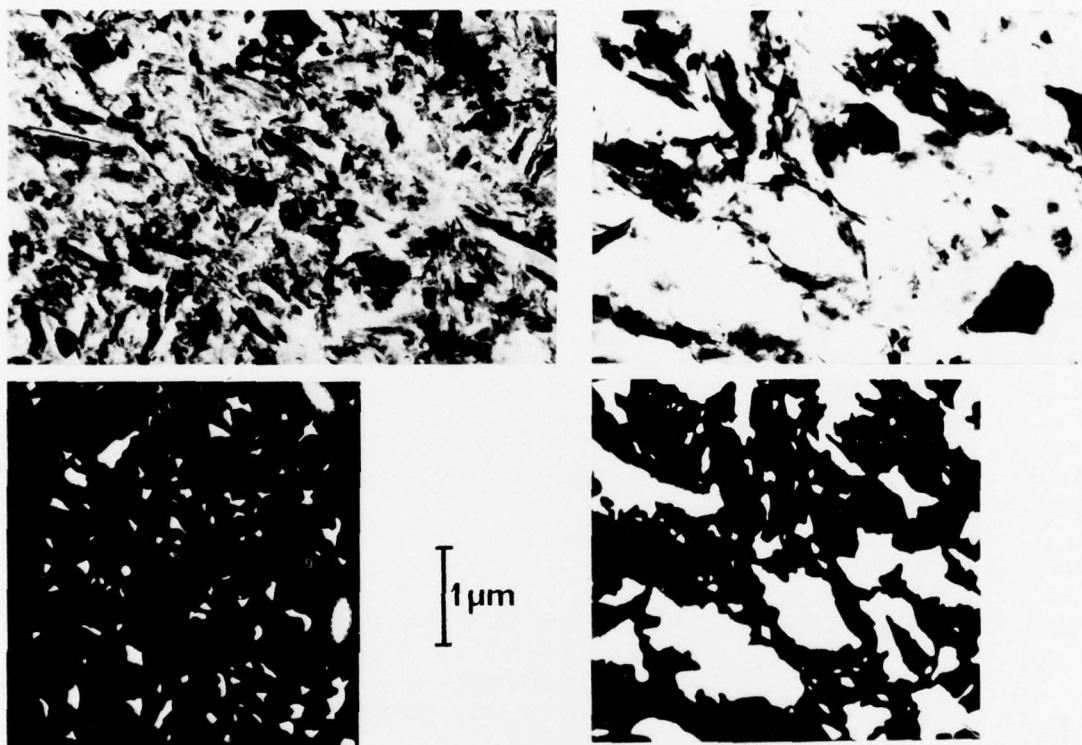CLAY MICROSTRUCTURE AND ICE NUCLEATION

Fig. 14   Micrographs (upper line) and drawn images of
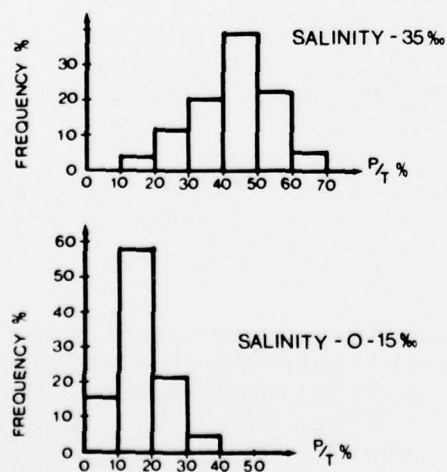   Skå-Edeby (left) and Lilla Edet clay samples.

Fig. 15   P/T versus salinity of the water in which the clays
          were formed. The histograms were based on several
          hundred micrographs.

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CONCLUSIONS

The investigation shows that the content of unfrozen water
is larger when the particle aggregates are small and
fairly porous but closely spaced than if the aggregates
are large and dense and separated by large pores. This is
because the microstructural constitution determines to what
extent the integrated mineral surface is exposed to the
pore water. Thus, the specific surface area is not a suffi-
cient measure of the content of unfrozen water. It should
be corrected with reference to the degree of aggregation.

A practical consequence of this investigation is that deter-
mination of the content of unfrozen water of structured
soils must be made by using soil samples with a defined
state: undisturbed or remoulded. The latter includes all
cases where the soil has a freeze history (previously frozen
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